

Claims

What is claimed is:

1. A method of producing a powder by a continuous reduction reaction, wherein said powder is a refractory metal powder, a valve metal powder, a refractory metal alloy powder, a valve metal alloy, a refractory metal suboxide powder, or a valve metal suboxide powder, comprising:
 - a) providing a refractory or valve metal oxide as a continuous feed or segments of continuous feed;
 - b) contacting said refractory or valve metal oxide feed with a reducing agent to create a static or dynamically formed mixture;
 - c) reducing said refractory or valve metal oxide feed in a reaction zone by heating said mixture in a reaction vessel to create a highly exothermic reaction, said exothermic reaction being triggered by heating said mixture to an ignition temperature and/or by adding a further reagent or catalyst; and
 - d) recovering a high surface area powder substantially free of impurities.
2. The method of claim 1 wherein said reducing agent is selected from the group consisting of magnesium, calcium, and aluminum.
3. The method of claim 1 wherein said reducing agent is in solid form in said mixture.
4. The method of claim 1 wherein said refractory or valve metal oxide feed is in solid form in said mixture.
5. The method of claim 1 wherein said mixture is formed prior to being fed into said reaction zone.
6. The method of claim 1 wherein said mixture is formed within said reaction zone.
7. The method of claim 1 wherein said reaction vessel is a vertical tube furnace.
8. The method of claim 1 wherein said refractory or valve metal oxide feed is selected from the group consisting of tantalum pentoxide, niobium pentoxide, niobium suboxide, zirconium dioxide, tungsten trioxide, chromium trioxide, molybdenum trioxide, titanium dioxide, vanadium pentoxide and niobium oxide or mixtures thereof.
9. The method of claim 1 wherein said refractory metal powder, said valve metal powder, said refractory metal alloy powder, or said valve metal alloy powder

consists essentially of tantalum, niobium, molybdenum, tungsten, vanadium, chromium, titanium or combinations thereof.

10. The method of claim 1 wherein said refractory metal suboxide powder or valve metal suboxide powder is selected from the group consisting of niobium suboxide, tungsten suboxide, molybdenum suboxide, vanadium suboxide, titanium suboxide, and chromium suboxide.
11. The method of claim 1 wherein the temperature in said reaction vessel is less than or equal to the melting point of said refractory or valve metal oxide feed.
12. The method of claim 1 wherein said exothermic reaction is self-sustaining.
13. The method of claim 1 wherein said powder further comprises agglomerates, said agglomerates having a substantially uniform particle size distribution.
14. The method of claim 1 wherein said powder further comprises agglomerates, said agglomerates having a bimodal particle size distribution.
15. The method of claim 1 further comprising adjusting at least one process parameter to control the chemical and physical properties of said powder, wherein said process parameter is selected from the group consisting of reagent feed rates, ignition temperature, steady state energy supply, reagent particle size, reducing agent stoichiometry, and inert carrier gas flow rate.
16. The method of claim 1 wherein said reducing agent in said mixture is provided in an amount substantially equal to the stoichiometric quantity required to react with said refractory or valve metal oxide feed.
17. The method of claim 1 further comprising forming said powder into pellets at an appropriate sintering temperature.
18. The method of claim 17 further comprising forming said sintered pellets into electrolytic capacitors.
19. A capacitor as produced by the method of claim 18 having a reduced solid capacitor capacitance change with increased formation voltage, an improved wet to solid capacitance recovery, and an improved equivalent series resistance.
20. A pellet as produced by the method of claim 17 having a pore size distribution relatively unchanged after anodization.

21. The method of claim 1 wherein the recovering step (d) further comprises agglomerating and/or deoxidizing.
22. The method of claim 21 further comprising forming said powder into pellets at an appropriate sintering temperature.
23. The method of claim 22 further comprising forming said sintered pellets into electrolytic capacitors.
24. A capacitor as produced by the method of claim 23 having a reduced solid capacitor capacitance change with increased formation voltage, an improved wet to solid capacitance recovery, and an improved equivalent series resistance.
25. A pellet as produced by the method of claim 22 having a pore size distribution relatively unchanged after anodization.
26. A high purity powder of essentially uniform particle size and substantially free of impurities produced by the method of claim 21, wherein said powder is a refractory metal powder.
27. The powder of claim 26 wherein said powder is essentially tantalum and said powder has an average surface area of about 1-5 m²/gm and a bulk density of about 15-35 gm/in³.
28. The powder of claim 26 wherein said powder is essentially niobium and said powder has an average surface area of about 1-7 m²/gm.
29. A high purity powder of essentially uniform particle size and substantially free of impurities produced by the method of claim 1, wherein said powder is a refractory metal powder, a valve metal powder, a refractory metal alloy powder, a valve metal alloy powder, a refractory metal oxide powder, or a valve metal oxide powder.
30. The powder of claim 29 further comprising agglomerates wherein said agglomerates have a substantially uniform particle size distribution.
31. The powder of claim 29 further comprising agglomerates wherein said agglomerates have a bimodal particle size distribution.
32. The powder of claim 29 wherein said refractory metal powder, said valve metal powder, said refractory metal alloy powder, or said valve metal alloy powder is selected from the group consisting essentially of tantalum, niobium, molybdenum, tungsten, vanadium, chromium, titanium, and combinations thereof.

33. The powder of claim 29 wherein said refractory metal oxide powder and said valve metal oxide powder is selected from the group consisting niobium suboxide, tungsten suboxide, molybdenum suboxide, vanadium suboxide, titanium suboxide, and chromium suboxide.
34. The powder of claim 29 wherein said powder is essentially tantalum and said powder has an average surface area of about $1 - 40 \text{ m}^2/\text{gm}$ and a bulk density of about $15-35 \text{ gm/in}^3$.
35. The powder of claim 29 wherein said powder is essentially niobium and said powder has an average surface area of about $1 - 40 \text{ m}^2/\text{gm}$.
36. The powder of claim 29 wherein said powder has an oxygen content of less than 6.0 wt %.
37. A high purity powder wherein said powder is a refractory metal powder, a valve metal powder, a refractory metal alloy powder, a valve metal alloy powder, a refractory metal oxide powder, or a valve metal oxide powder, comprising particles of essentially uniform particle size and substantially free of impurities.
38. The powder of claim 37 further comprising agglomerates wherein said agglomerates have a substantially uniform particle size distribution.
39. The powder of claim 37 further comprising agglomerates wherein said agglomerates have a bimodal particle size distribution.
40. The powder of claim 37 wherein said refractory metal powder, said valve metal powder, said refractory metal alloy powder, or said valve metal alloy powder has an oxygen content of less than 6.0 wt %.
41. The powder of claim 37 wherein said refractory metal powder, said valve metal powder, said refractory metal alloy powder, or said valve metal alloy powder is selected from the group consisting of tantalum, niobium, molybdenum, tungsten, vanadium, chromium, titanium, and combinations thereof.
42. The powder of claim 37 wherein said refractory metal oxide powder or said valve metal oxide powder is selected from the group consisting of niobium suboxide, tungsten suboxide, molybdenum suboxide, vanadium suboxide, titanium suboxide, and chromium suboxide.

43. The powder of claim 37 wherein said impurities is selected from the group consisting of metallics, halides, and alkalis.
44. The powder of claim 37 wherein said powder is essentially tantalum and said powder has an average surface area of about 1-40 m²/gm and a bulk density of about 15-35 gm/in³.
45. The powder of claim 44 wherein said average surface area is about 1-5 m²/gm.
46. The powder of claim 37 wherein said powder is essentially niobium and said powder has an average surface area of about 1-40 m²/gm.
47. The powder of claim 46 wherein said average surface area is about 1-7 m²/gm.
48. A pellet comprising the powder of claim 37 having a pore size distribution relatively unchanged after anodization.
49. A capacitor comprising the powder of claim 37 having a reduced solid capacitor capacitance change with increased formation voltage, an improved wet to solid capacitance recovery, and an improved equivalent series resistance.

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